

# इंटरनेट

# मानक

## Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 8954 (1978): Edifenphos, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



BLANK PAGE



**IS : 8954 - 1978**

*Indian Standard*

**SPECIFICATION FOR  
EDIFENPHOS, TECHNICAL**

**(First Reprint NOVEMBER 1999)**

**UDC 632.952 EDI**

**© BIS 1979**

**BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002**

**AMENDMENT NO. 3    DECEMBER 1995**  
**TO**  
**IS 8954 : 1978    SPECIFICATION FOR EDIFENPHOS,**  
**TECHNICAL**

( Page 4, Table 1, col 5 ) — Substitute 'IS 6940 : 1982\*' for 'IS : 6940 - 1973\*'.

( Page 4, Table 1, foot-note with ' \* ' mark ) — Add '( first revision )' at the end of text.

( Page 5, clause 3.1 ) — Substitute 'IS : 8190 ( Part 2 ) - 1988\*' for 'IS : 8190 ( Part II ) - 1976\*'.

( Page 5, foot-note with ' \* ' mark ) — Add '( second revision )' at the end of text.

( Page 5, clause 4.1 ) — Substitute the following for the existing clause:

'Representative samples of the material shall be drawn according to IS 10946 : 1984.'

( Page 6, clause 5.2 ) — Substitute 'IS 1070 : 1992\*' for 'IS : 1070 - 1977\*'.

( Page 6, foot-note with ' \* ' mark ) — Substitute 'Reagent grade water ( third revision )' for the existing title.

[ Page 9, clause A-2.3 ( see also Amendment No. 2 ) ] — Substitute the following for the existing formula:

$$\text{Edifenphos content, percent by mass} = \left( \frac{SA}{M_1} - \frac{B}{M_2} \right) \times 0.1553 \times 100$$

**AMENDMENT NO. 4 DECEMBER 2003**  
**TO**  
**IS 8954 : 1978 SPECIFICATION FOR EDIFENPHOS,**  
**TECHNICAL**

( Page 6, Appendix A ) — Substitute the following for the existing:

**ANNEX A**  
**[ Table 1, Item (i) ]**

**DETERMINATION OF EDIFENPHOS CONTENT**

**A-0 GENERAL**

Either of the two methods, namely, gas chromatographic method or iodine method may be used for determination of Edifenphos content. However, in case of dispute, gas chromatographic method shall be the referee method.

**A-1 GAS CHROMATOGRAPHIC METHOD**

**A-1.1 Principle of the Method**

The method consists of injecting a sample with an internal standard in a known proportion into a gas chromatograph and determining the area under peak. The area under the peak is proportional to the mass of the sample. By comparison of this area with that of the standard, the percentage purity of the sample is determined.

**A-1.2 Apparatus**

**A-1.2.1 Gas Liquid Chromatograph ( GLC )** — Equipped with a flame ionization detector ( FID ) and coupled to a printer plotter-cum-integrator.

Detector temperature	300°C
Injection temperature	250°C
Oven temperature	195°C
Carrier gas flow rate ( N <sub>2</sub> ), ml/min	30
Hydrogen, ml/min	30
Air, ml/min	300
Attenuation	2 ↑ 9

## **Amend No. 4 to IS 8954 : 1978**

**A-1.2.2 Column** — Consisting of 180-cm glass tubing of 4.0 mm outer diameter packed with 2 percent OV-101 on Gaschrome Q 100-120 Mesh.

**A-1.2.3 Micro Syringe** — 10  $\mu$ l capacity.

### **A-1.3 Reagents**

#### **A-1.3.1 Acetone AR Grade**

**A-1.3.2 Internal Standard Solution** — Dioctyl adipate ( DOA ) free from any impurity likely to interfere with Edifenphos under the chromatographic conditions.

**A-1.3.3 Standard Reference Edifenphos** — Edifenphos standard of known purity.

### **A-1.4 Procedure**

#### **A-1.4.1 Preparation of Internal Standard**

Dissolve 1.2 g of dioctyl adipate in 100 ml of acetone.

#### **A-1.4.2 Preparation of Sample Solution**

Weigh a sample containing about 0.25 g of Edifenphos in 25-ml volumetric flask and add to it 20 ml of the internal standard solution. Dilute up to the mark with acetone and shake well to homogenize.

#### **A-1.4.3 Preparation of Standard Solution**

Weigh accurately about 0.25 g of Edifenphos standard of known purity in 25-ml volumetric flask and add to it 20 ml of 1.2 percent Dioctyl adipate (DOA) in acetone solution and dilute up to the mark with acetone and shake well to homogenize.

#### **A-1.4.4 Analysis of Sample**

Inject 3  $\mu$ l of standard solution (*see A-1.4.3*) and sample solution (*see A-1.4.2*) to GLC column set up at the prescribed operating conditions (*see A-1.2.1*) and record the GLC charts. Obtain the peak area ratios of Edifenphos, namely, dioctyl adipate from the GLC charts of the Edifenphos reference standard solution (*see A-1.4.3*) and sample solution (*see A-1.4.2*).

### A-1.5 Calculation

$$\text{Edifenphos content, percent by mass} = \frac{A_3 \times A_2 \times M_1 \times P}{A_4 \times A_1 \times m_2}$$

where

- $A_1$  = area of the Edifenphos peak in the standard solution;
- $A_2$  = area of the Edifenphos peak in the sample solution;
- $A_3$  = area of the internal standard peak in the standard solution;
- $A_4$  = area of the internal standard peak in the sample solution;
- $m_1$  = mass, in g, of the Edifenphos standard (A-1.4.3);
- $m_2$  = mass, in g, of the sample taken for the test (A-1.4.2); and
- $P$  = percentage purity of the Edifenphos standard.

## A-2 IODOMETRIC METHOD

### A-2.1 Reagent

A-2.1.1 *Standard Sulphuric Acid* — 3 to 4 N.

A-2.1.2 *Sodium Hydrogen Carbonate*

A-2.1.3 *Sodium Hydroxide Pellets*

A-2.1.4 *Standard Iodine* — 0.1 N.

A-2.1.5 *Phenolphthalein Indicator Solution* — 0.1 percent.

A-2.1.6 *Ethanol*

A-2.1.7 *Starch* — 1 percent.

A-2.1.8 *Standard Sodium Thiosulphate* — 0.1 N.

### A-2.2 Determination of Total Thiophenol Content

Weigh accurately about 0.5 g of the sample into a 250-ml three neck round bottom flask with ground glass joint and dissolve it in 30-ml ethyl alcohol. Add 20 ml distilled water and 5 g of sodium hydroxide pellets. Heat the mixture to reflux on a heating mantle using a water condenser in an atmosphere of nitrogen for 4 h. Add few millilitre of ethyl alcohol, if required. After reflux wash the condenser with 10 ml of ethyl alcohol. Cool the reaction mixture to room temperature and transfer quantitatively to a 250-ml volumetric flask using 80 ml



#### Amend No. 4 to IS 8954 : 1978

of ethyl alcohol. Make up to the mark with distilled water. Shake well to homogenize the solution. Pipette out 50 ml of solution into a 250-ml B 24 joint conical flask. Add 2-3 drops of phenolphthalein indicator and acidify with 3 - 4N sulphuric acid. If the solution turns milky add ethyl alcohol until the solution becomes clear. Add a pinch of sodium hydrogen carbonate to confirm that the solution is acidic. Add 20 ml of standard 0.1N iodine solution and titrate against standard 0.1N sodium thiosulphate solution using starch solution as an indicator. The end point is blue to colourless.

**A-2.2.1** Carry out the blank titration for 20 ml of iodine solution.

#### **A-2.2.2** *Determination of Free Thiophenol Content*

Weigh accurately about 0.5 g of the sample into a 250-ml stoppered conical flask and dilute it with 40 ml of ice cooled ethyl alcohol. Add 20 ml of ice cooled distilled water, 5 ml of 3-4 N sulphuric acid and a pinch of sodium hydrogen carbonate. Titrate it immediately against 0.1N standard Iodine solution. The end point is the appearance of blue colour.

#### **A-2.3** Calculation

$$\text{Ediphenphos content, } \frac{m}{m} = \frac{5(V_0 - V_1)}{M} \times \frac{V_2}{m} \times 15.52 \times N$$

where

$V_0$  = volume, in ml, of standard 0.1N sodium thiosulphate solution required for 20 ml of iodine solution (*see A-2.2.1*);

$V_1$  = volume, in ml, of standard 0.1N sodium thiosulphate solution required for the estimation of total thiophenol content (*see A-2.2*);

$V_2$  = volume, in ml, of standard 0.1N iodine solution consumed for free thiophenol equivalent to standard 0.1N sodium thiosulphate solution (*see A-2.2.2*);

$M$  = mass, in g, of the sample taken for the estimation of the total thiophenol content;

$m$  = mass, in g, of the sample taken for the estimation of free thiophenol content; and

$N$  = normality of standard 0.1N sodium thiosulphate solution.

(FAD 1)

**AMENDMENT NO. 4 DECEMBER 2003**  
**TO**  
**IS 8954 : 1978 SPECIFICATION FOR EDIFENPHOS,**  
**TECHNICAL**

( Page 6, Appendix A ) — Substitute the following for the existing:

**ANNEX A**  
**[ Table 1, Item (i) ]**

**DETERMINATION OF EDIFENPHOS CONTENT**

**A-0 GENERAL**

Either of the two methods, namely, gas chromatographic method or iodine method may be used for determination of Edifenphos content. However, in case of dispute, gas chromatographic method shall be the referee method.

**A-1 GAS CHROMATOGRAPHIC METHOD**

**A-1.1 Principle of the Method**

The method consists of injecting a sample with an internal standard in a known proportion into a gas chromatograph and determining the area under peak. The area under the peak is proportional to the mass of the sample. By comparison of this area with that of the standard, the percentage purity of the sample is determined.

**A-1.2 Apparatus**

**A-1.2.1 Gas Liquid Chromatograph ( GLC )** — Equipped with a flame ionization detector ( FID ) and coupled to a printer plotter-cum-integrator.

Detector temperature	300°C
Injection temperature	250°C
Oven temperature	195°C
Carrier gas flow rate ( N <sub>2</sub> ), ml/min	30
Hydrogen, ml/min	30
Air, ml/min	300
Attenuation	2 ↑ 9

## **Amend No. 4 to IS 8954 : 1978**

**A-1.2.2 Column** — Consisting of 180-cm glass tubing of 4.0 mm outer diameter packed with 2 percent OV-101 on Gaschrome Q 100-120 Mesh.

**A-1.2.3 Micro Syringe** — 10  $\mu$ l capacity.

### **A-1.3 Reagents**

**A-1.3.1 Acetone AR Grade**

**A-1.3.2 Internal Standard Solution** — Dioctyl adipate ( DOA ) free from any impurity likely to interfere with Edifenphos under the chromatographic conditions.

**A-1.3.3 Standard Reference Edifenphos** — Edifenphos standard of known purity.

### **A-1.4 Procedure**

**A-1.4.1 Preparation of Internal Standard**

Dissolve 1.2 g of dioctyl adipate in 100 ml of acetone.

**A-1.4.2 Preparation of Sample Solution**

Weigh a sample containing about 0.25 g of Edifenphos in 25-ml volumetric flask and add to it 20 ml of the internal standard solution. Dilute up to the mark with acetone and shake well to homogenize.

**A-1.4.3 Preparation of Standard Solution**

Weigh accurately about 0.25 g of Edifenphos standard of known purity in 25-ml volumetric flask and add to it 20 ml of 1.2 percent Dioctyl adipate (DOA) in acetone solution and dilute up to the mark with acetone and shake well to homogenize.

**A-1.4.4 Analysis of Sample**

Inject 3  $\mu$ l of standard solution (see A-1.4.3) and sample solution (see A-1.4.2) to GLC column set up at the prescribed operating conditions (see A-1.2.1) and record the GLC charts. Obtain the peak area ratios of Edifenphos, namely, dioctyl adipate from the GLC charts of the Edifenphos reference standard solution (see A-1.4.3) and sample solution (see A-1.4.2).

### A-1.5 Calculation

$$\text{Edifenphos content, percent by mass} = \frac{A_3 \times A_2 \times M_1 \times P}{A_4 \times A_1 \times m_2}$$

where

- $A_1$  = area of the Edifenphos peak in the standard solution;
- $A_2$  = area of the Edifenphos peak in the sample solution;
- $A_3$  = area of the internal standard peak in the standard solution;
- $A_4$  = area of the internal standard peak in the sample solution;
- $m_1$  = mass, in g, of the Edifenphos standard (A-1.4.3);
- $m_2$  = mass, in g, of the sample taken for the test (A-1.4.2); and
- $P$  = percentage purity of the Edifenphos standard.

## A-2 IODOMETRIC METHOD

### A-2.1 Reagent

A-2.1.1 *Standard Sulphuric Acid* — 3 to 4 N.

A-2.1.2 *Sodium Hydrogen Carbonate*

A-2.1.3 *Sodium Hydroxide Pellets*

A-2.1.4 *Standard Iodine* — 0.1 N.

A-2.1.5 *Phenolphthalein Indicator Solution* — 0.1 percent.

A-2.1.6 *Ethanol*

A-2.1.7 *Starch* — 1 percent.

A-2.1.8 *Standard Sodium Thiosulphate* — 0.1 N.

### A-2.2 Determination of Total Thiophenol Content

Weigh accurately about 0.5 g of the sample into a 250-ml three neck round bottom flask with ground glass joint and dissolve it in 30-ml ethyl alcohol. Add 20 ml distilled water and 5 g of sodium hydroxide pellets. Heat the mixture to reflux on a heating mantle using a water condenser in an atmosphere of nitrogen for 4 h. Add few millilitre of ethyl alcohol, if required. After reflux wash the condenser with 10 ml of ethyl alcohol. Cool the reaction mixture to room temperature and transfer quantitatively to a 250-ml volumetric flask using 80 ml

#### **Amend No. 4 to IS 8954 : 1978**

of ethyl alcohol. Make up to the mark with distilled water. Shake well to homogenize the solution. Pipette out 50 ml of solution into a 250-ml B 24 joint conical flask. Add 2-3 drops of phenolphthalein indicator and acidify with 3 - 4N sulphuric acid. If the solution turns milky add ethyl alcohol until the solution becomes clear. Add a pinch of sodium hydrogen carbonate to confirm that the solution is acidic. Add 20 ml of standard 0.1N iodine solution and titrate against standard 0.1N sodium thiosulphate solution using starch solution as an indicator. The end point is blue to colourless.

**A-2.2.1** Carry out the blank titration for 20 ml of iodine solution.

#### **A-2.2.2 Determination of Free Thiophenol Content**

Weigh accurately about 0.5 g of the sample into a 250-ml stoppered conical flask and dilute it with 40 ml of ice cooled ethyl alcohol. Add 20 ml of ice cooled distilled water, 5 ml of 3-4 N sulphuric acid and a pinch of sodium hydrogen carbonate. Titrate it immediately against 0.1N standard Iodine solution. The end point is the appearance of blue colour.

#### **A-2.3 Calculation**

$$\text{Ediphenphos content, } \frac{\text{percent, } m/m}{M} = \frac{5 (V_0 - V_1)}{M} \times \frac{V_2}{m} \times 15.52 \times N$$

where

$V_0$  = volume, in ml, of standard 0.1N sodium thiosulphate solution required for 20 ml of iodine solution (see A-2.2.1);

$V_1$  = volume, in ml, of standard 0.1N sodium thiosulphate solution required for the estimation of total thiophenol content (see A-2.2);

$V_2$  = volume, in ml, of standard 0.1N iodine solution consumed for free thiophenol equivalent to standard 0.1N sodium thiosulphate solution (see A-2.2.2);

$M$  = mass, in g, of the sample taken for the estimation of the total thiophenol content;

$m$  = mass, in g, of the sample taken for the estimation of free thiophenol content; and

$N$  = normality of standard 0.1N sodium thiosulphate solution.

( FAD 1 )

AMENDMENT NO. 1    DECEMBER 1980  
TO  
IS:8954-1978   SPECIFICATION FOR EDIFENPHOS, TECHNICAL

Corrigendum

(Page 9, clause A-2.3, formula) - Substitute the following for the existing formula:

$$\begin{array}{l} \text{'Edifenphos content,} \\ \text{percent by mass} \end{array} = \left[ \frac{5A}{M_1} - \frac{B}{M_2} \right] \times 1.552 \times 0.935$$

(AFCD 6)

**AMENDMENT NO. 2 JULY 1990**  
**TO**  
**IS 8954 : 1978 SPECIFICATION FOR EDIFENPHOS,**  
**TECHNICAL**

( *Page 9, clause A-2.3* ) — Substitute the following for the existing formula:

$$\text{'Edifenphos content, percent by mass'} = \left( \frac{5A}{M_1} - \frac{B}{M_2} \right) \times 0.1553 \times 100 \times 0.935'$$

( FADC 1 )

Reprography Unit, BIS, New Delhi, India

( FAD 1 )

Reprography Unit, BIS, New Delhi, India

**AMENDMENT NO. 3 DECEMBER 1995**  
**TO**  
**IS 8954 : 1978 SPECIFICATION FOR**  
**EDIFENPHOS, TECHNICAL**

( *Page 4, Table 1, col 5* ) — Substitute 'IS 6940 : 1982\*' for 'IS : 6940 - 1973\*'.

( *Page 4, Table 1, foot-note with ' \* ' mark* ) — Add '( *first revision* )' at the end of text.

( *Page 5, clause 3.1* ) — Substitute 'IS : 8190 ( Part 2 ) - 1988\*' for 'IS : 8190 ( Part II ) - 1976\*'.

( *Page 5, foot-note with ' \* ' mark* ) — Add '( *second revision* )' at the end of text.

( *Page 5, clause 4.1* ) — Substitute the following for the existing clause:

'Representative samples of the material shall be drawn according to IS 10946 : 1984.'

( *Page 6, clause 5.2* ) — Substitute 'IS 1070 : 1992\*' for 'IS : 1070 - 1977\*'.

( *Page 6, foot-note with ' \* ' mark* ) — Substitute 'Reagent grade water ( *third revision* )' for the existing title.

[ *Page 9, clause A-2.3 ( see also Amendment No. 2 )* ] — Substitute the following for the existing formula:

$$\begin{array}{l} \text{Edifenphos content,} \\ \text{percent by mass} \end{array} = \left( \frac{5A}{M_1} - \frac{B}{M_2} \right) \times 0.1553 \times 100$$